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## ***Regolith Volatile Characterization (RVC) in RESOLVE***

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### **Abstract**

Resource investigation in the lunar poles is of importance to the potential impact of in-situ resource utilization (ISRU). The RESOLVE project developed a payload to investigate the permanently shadowed areas of the lunar poles and demonstrate ISRU technology. As a part of the RESOLVE project, the regolith volatile characterization (RVC) subsystem was designed to examine the release of volatiles from sample cores. The test sample was heated in the reactor to release the volatiles where they were analyzed with gas chromatography. Subsequently, the volatile sample was introduced into the lunar water resource demonstration (LWRD) subsystem where the released hydrogen and water were selectively captured.

The objective of the Regolith Volatile Characterization (RVC) subsystem was to heat the crushed core sample and determine the desorption of volatile species of interest. The RVC subsystem encompasses the reactor and the system for volatile analysis. The system was designed to analyze H<sub>2</sub>, He, CO, CO<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub>, CH<sub>4</sub>, H<sub>2</sub>S and H<sub>2</sub>O. The GC chosen for this work is a Siemens MicroSAM process GC with 3 columns and 8 TCD detectors. Neon was chosen as the carrier gas to enhance the analysis of hydrogen and helium. The limit of detection for the gases is ~1000ppm for H<sub>2</sub>, CO, CO<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub> and H<sub>2</sub>S. The limit of detection for CH<sub>4</sub> is ~4000ppm and the water limit of detection is ~10000 ppm with a sample analysis time of 2-3 minutes. These values (with the exception of water and H<sub>2</sub>S) were determined by dilution of a six gas mixture from Scott Gas (5% CO<sub>2</sub>, CO, O<sub>2</sub>, N<sub>2</sub>, 4% CH<sub>4</sub> and H<sub>2</sub>) using mass flow controllers (MFCs). Water was calibrated at low levels using an in house relative humidity (RH) generator. H<sub>2</sub>S and high concentrations of H<sub>2</sub> were calibrated by diluting a pure stream of gas with MFCs. Higher concentrations of N<sub>2</sub> and O<sub>2</sub> were calibrated using Air again diluting with MFCs.

There were three modification goals for the GC in EBU2 that would allow this process GC to be used in the field demo for RESOLVE. The first modification was to decrease the weight associated with the GC, this included eliminating the explosion proof case (Figure 1) and replacing it with a lightweight case as well as using an on board COPV tank for the neon carrier gas. The next goal was to add a second oven for the molecular sieve column to allow for dual temperature control during GC operation; the separation of hydrogen and helium is optimum at lower temperatures while the water analysis required higher temperatures creating a competing design requirement. The second oven also allows a lower limit of detection for water quantification and avoids the possibility of water condensing in the GC which could ruin the column characteristics. The final goal was to modify the column arrangement to optimize the system for our specific application. Figure 2 shows the internal details of the module optimized for our field application. The modifications and performance of the gas analysis system will be discussed in detail.



Figure 1. Internal components of the MicroSAM GC with original explosion proof housing

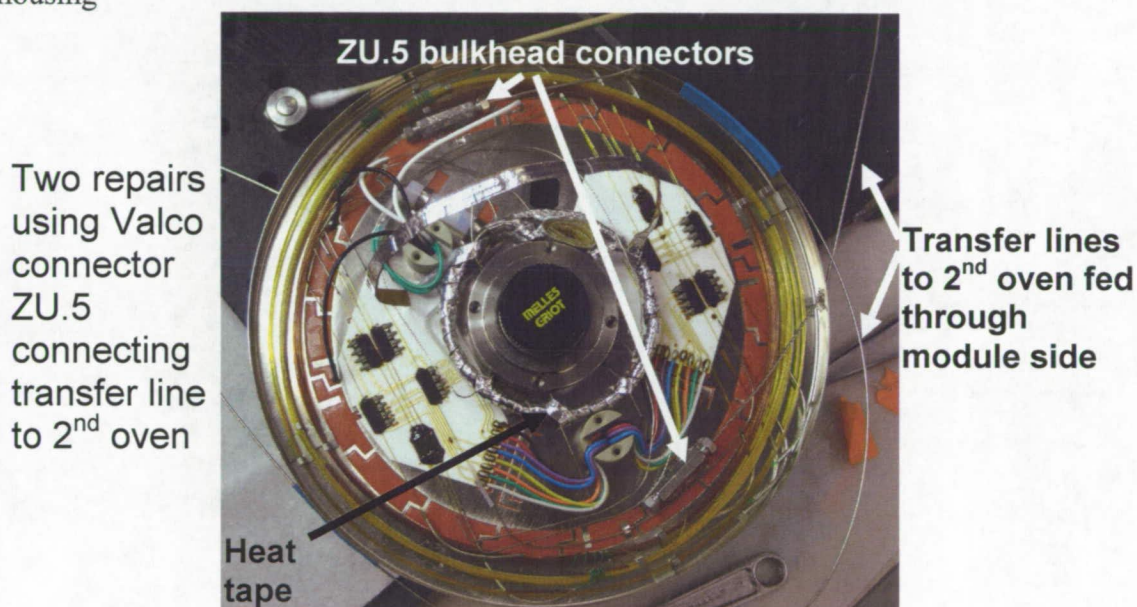


Figure 2. Module modification for field testing

### **General Design Selections**

The design of the system was strongly driven by the field site test objectives. There were several differences between the lunar application and terrestrial field application that influenced our design. Initializing the heating of the soil with atmospheric pressure in the oven affected the abundance of nitrogen and oxygen present in our sample stream. The lack of hydrogen present in the terrestrial environment also affected our design. Both factors affected the sample size, the sampling frequency and the range of our calibrations that were performed in the lab prior to field site testing. Modifications to the operating conditions allowed the GC to be tailored for lab and field site testing.

In the operational procedure of RESOLVE, there were several choices that determined the way the system was programmed for automated operation. One of the first choices that needed to be made was deciding when to transfer gas from the reactor to the surge tank. For this generation of the GC, a minimum of 1psi over atmospheric pressure is required in order for the GC to obtain a sample. This requirement played a role in the decision of when to transfer to the surge tank. If transferring to the surge tank resulted in a pressure lower than atmospheric pressure, no further GC samples could be taken. From the GC point of view, it was desirable to build up pressure in the reactor and sample continuously. This approach would also minimize the error introduced into the system due to water vapor generation during transfer. The drawback to this approach was that if a compound reached saturation at a specific temperature and was not transferred to the surge tank, no further volatilization of that compound would take place and information on the correlation of the volatilization with temperature would be lost. However, given the time limitation imposed on the overall system operation, the pressure build up approach was agreed upon by the various subsystems. Further work showed that no compounds in our system reached saturation during heating and in fact the water generation from the soil was much slower than initially expected. A very slow heating profile along with the ability to sample at sub-atmospheric pressures would be required to really understand the binding of the water to the regolith. Although this was a goal of the project initially, time requirements for the field demonstration would not allow for this goal to be met.

The limit of detection for the gases is ~1000ppm for  $H_2$ , CO,  $CO_2$ ,  $N_2$ ,  $O_2$  and  $H_2S$ . The limit of detection for  $CH_4$  is ~4000ppm and the water limit of detection is ~10000ppm. These values (with the exception of water and  $H_2S$ ) were determined by dilution of a six gas mixture from Scott Gas (5%  $CO_2$ , CO,  $O_2$ ,  $N_2$ , 4%  $CH_4$  and  $H_2$ ) using mass flow controllers (MFCs). Water was calibrated at low levels using an in house relative humidity (RH) generator and  $H_2S$  and high concentrations of  $H_2$  were calibrated by diluting a pure stream of gas with MFCs. Higher concentrations of  $N_2$  and  $O_2$  were calibrated using Air again diluting with MFCs. Figure 3 shows GC spectra of various gas samples from calibration and integrated testing. The first two spectra are from higher level calibration runs for air and hydrogen, the last spectra is from an integrated test run showing carbon dioxide and water.



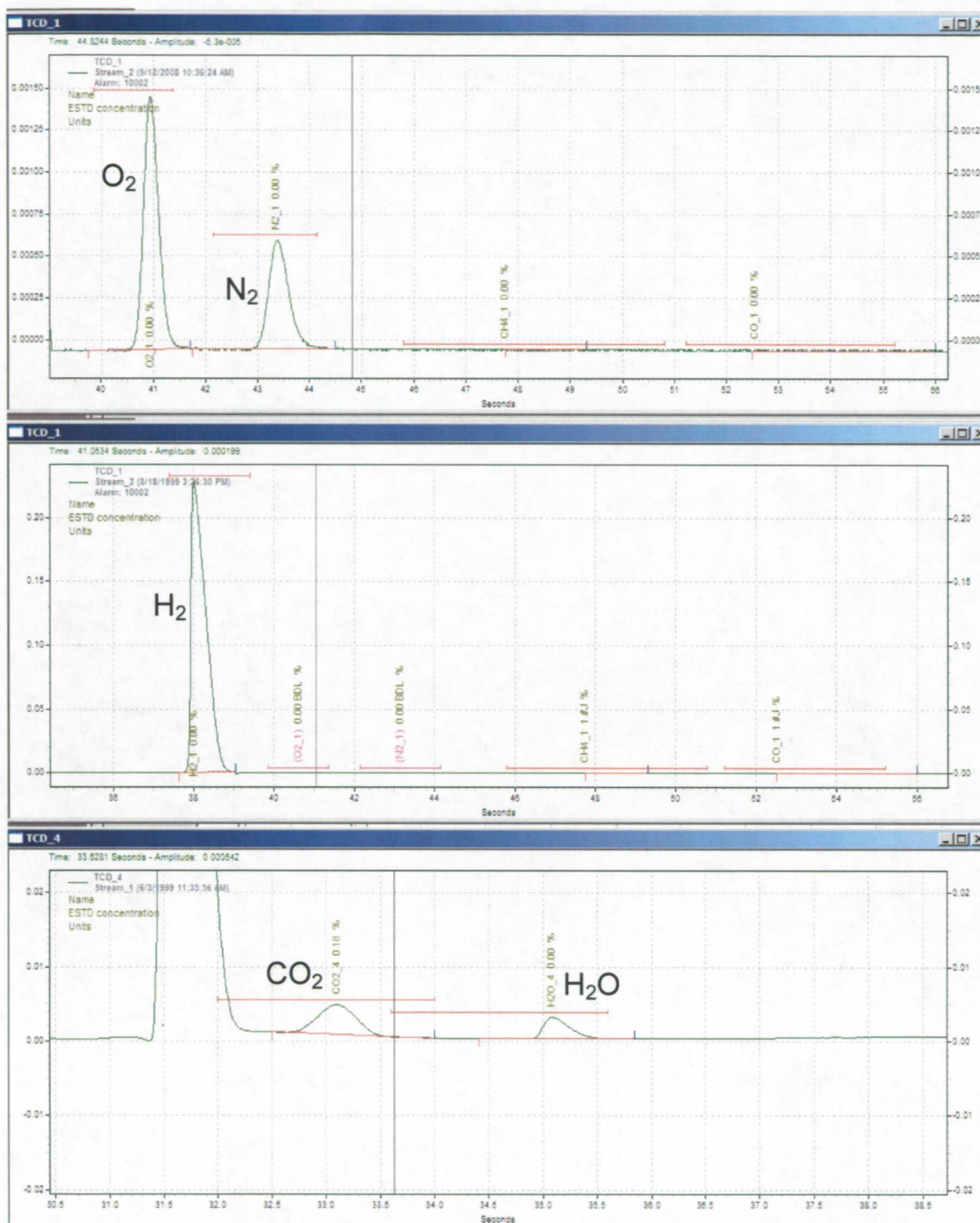


Figure 3. Examples of GC spectra from calibration and integrated testing

Converting the GC data (which is obtained in ppm or percent) to mass is done by calculating the total amount of gas in the reactor at the time the sample was taken. This is done using the ideal gas law ( $PV=nRT$ ). The pressure was measured by the pressure transducer in the reactor and the control point for the oven temperature was used as the temperature. The temperature measurement introduces some error because the temperature reading is on an external point in the reactor as there are no temperature

readings inside the reactor. Increased accuracy could be achieved with an accurate internal temperature reading.

### **HI Testing**

Once the hardware arrived in Hilo, the hardware was assembled, tested, and integrated with the rover. Once installed, the chemical plant was left in place in the rover chassis for the remainder of the Hawaii testing. Six integrated tests were performed in Hawaii over a seven day period. The tests conducted and general results will be presented in more detail in the following paragraphs.

The first fully integrated test with the rover involved the testing of day 1 operations based on the Hawaii test plan. This test involved testing a single ¼ core of pre-treated Tefra (dried to 1% water and sieved to < 1 mm) that was crushed using the NORCAT crusher. This was the second ¼ core from the full core that was drilled and captured on the previous evening. Sample had to be added from the first ¼ core to supplement the sample since a full core was not captured. The reactor was charged with the crushed sample and air. Autonomous computer control (MEC) and OPC control were utilized in the testing. Power from the GSE cart was used to perform all operations. Twelve GC samples were taken during the RVC operations, with reactor pressures ranging from 0.5 psig – 6.0 psig. The GC data collected for this test indicated that a small quantity of water was released during RVC operations. All chromatograms indicated the presence of oxygen, nitrogen, carbon dioxide, and water in the reactor.

The remaining tests were similar to the first test described previously with minor changes in the number of GC samples and the overall pressures in the reactor.

### **HI Test Results**

The same series of factors and assumptions were used when analyzing the Hawaii test data. A summary of data from the Hawaii testing can be found in Table 1.

Table 1. Summary of Hawaii testing results.

Date	GC analysis (mg water in reactor prior to 1 <sup>st</sup> transfer)	Final amount of water from GC (additional water generated during final GC run)	Amount of water lost during GC sampling as reactor is heating	Total amount of water quantified by GC prior to 1 <sup>st</sup> sample transfer to ST
11/5/08	46.5mg	47.8mg	21.8mg	69.9mg
11/6/08	45.0mg	47.3mg	23.9mg	71.2mg
11/8/08	42.7mg	45.7mg	13.3mg	59.0mg
11/9/08	44.6mg	44.6mg	N/A**	44.6mg

11/10/08	49.0mg	52.0mg	17.9mg	69.9mg
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\*\* 11/9/08 – Valco valve malfunction, premature transfer and no GC readings

The GC data again indicates that the water content of the pre-treated Tefra was much less than the anticipated 1%. However, the total water quantified prior to transfer was consistent with the data from the first integrated tests at Carnegie Mellon University, adding credibility to the data.

### **Laboratory H2 testing**

Laboratory testing with a doped reactor was done to simulate a lunar sample consisting of mainly hydrogen and water. The high concentration of hydrogen required an additional method to be created to avoid saturating the detector. Two GC methods were used in the laboratory testing, similar to the testing done in the field. The two methods were calibrated for high concentrations of hydrogen. The first method used had a smaller (100ms) injection, while the second method used was the 300ms injection method used in the field. Manual control of the GC was used in this testing. The calculations of the amount of hydrogen and water during these integrated tests were performed under the same conditions as the field testing.

The reactor was purged and then dosed with hydrogen. Prior to the heaters on the reactor being turned on, there was 0.0448g of hydrogen in the reactor. The final GC reading showed there was only 0.0119g of hydrogen in the reactor. Estimating the amount of hydrogen lost during GC sampling accounts for only ~10% of the sample. The remainder of the loss can be attributed to the reactor leaking. This conclusion is supported by the decrease in pressure over time, even with the reactor temperature increasing. If the reactor were sealed completely, the pressure in the reactor should have been ~79 psia from the hydrogen gas, and any water desorbed would have further increased the pressure. The data showed a final water reading of 0.0748g in the reactor prior to transfer to the LWRD subsystem. Similar tests results were obtained for various amounts of hydrogen. The reactor seals were corrupted by the soil leading to leaking seals in the system. While the system was not able to form a perfect seal, the integrated tests with hydrogen showed the capability to measure both hydrogen and helium during integrated tests.

### **Conclusion**

The integrated tests of the RVC system in RESOLVE demonstrate the ability to identify and quantitate gases of interest for lunar applications. Improvements to the system will enhance this ability and provide more accurate data for future field testing. The unique capabilities and engineering modifications made to the RVC lightweight GC can be used for many field testing applications demanding gas quantitation from a lightweight instrument.